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SYNTHESIS, DESIGNING, CHARACTERIZATION AND ANTI-MICROBIAL STUDIES OF N, N'-BIS (2 HYDROXYACETOPHENONE) ETHYLENEDIAMINE METAL (II) COMPLEXES

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Abstract
Tetradentate Schiffbase N, N'-bis [2-hydroxyacetophenone] ethylendiamine (OAcPh-en) were formed by the 2:1 molar condensation of 2-hydroxyacetophenone with ethylenediamine. Their Ni (II), Cu (II), Co (II), Mn (II), Fe (II) and Zn (II) diaquocomplexes (I) were synthesized. The neutral bidentate ligand derived from benzaldehyde and ethylenediamine or o-phenylenediamine in 1:1 molar ratio in alcoholic solution yield bis [benzylidene] ethylenediamine (Ben-en) or bis [benzylidene] o-phenylinediamine (Ben-opd) (II). Further reaction between (I) and (II) in alcohol yielded mixed ligand complexes. The characterization of these newly synthesized mixed-ligand complexes was done by elemental analysis, magnetic measurements, infrared spectra and uv-visible spectra, thermo gravimetric analysis, anti-microbial studies have also been under taken.
Keywords: Shiff Bases; Mixed ligand complexes; Antimicrobial Activity; Synthesis; Characterization.

1. Introduction

Ligand, a metal surrounded by a cluster of ions or molecule, is used for preparation of complex compounds named as Schiff bases [1]. Schiff bases have been widely used in many fields e.g., biological, inorganic, analytical and drug synthesis, as bidentate ligands in the field of coordination chemistry [2]. The chemistry of Schiff base metal complexes had been investigated extensively for the last seven decades leading to new synthetic routes variety of structures and their potential biological alications [2]. Tetra dentate Schiff bases are well known for their coordination with various metal ions, forming stable compounds [3]. Many symmetrical bis tetradentate Schiff bases of 1, 2-diamines with 0- hydroxy aldehydes/ketones have been prepared and studied intensively. However, much less attention has been focused on unsymmetrical tetradentate Schiff bases derived from 1, 2-diamines and different aldehydes/ketones. In particular those derived from aromatic 1, 2-diamines have been under-investigated [4], [5]. We prepared new mixed ligand complexes of the type (M (OAcPh-en) (Ben-en) H2O and (M (OAcPh-en) (Ben-opd) H2O where M=Ni (II), Cu (II), Co(II), Fe(II), Mn(II) and Zn(II). OAcPh-en = N, N'-bis (2-hydroxyacetophenone) ethylenediamine, Ben-en = bis (benzaldehydel) ethylenediamine, Ben-opd = bis (benzaldehyde) o-phenylenediamine. The complexes were characterized on the basis of physical properties, elemental analysis, infrared, uv-visible spectra, thermo gravimetric analysis and antimicrobial activities.

2. Material and methods

2.1 Synthesis of N, N'-bis [2-hydroxyacetophenone] ethylenediamine (OAcph-en)

The ligand OAcPh-en was synthesized by refluxing an ethanolic solution of 2-hydroxyacetophenone (OAcPh) and ethylenediamine (en) (2:1 molar ratio) for one hour. The reaction mixture was cooled and a bright yellow crystalline solid of N, N'- bis (2-hydroxyacetophenone) ethylenediamine (OAcPh-en) separated [6]. The solid was filtered off and recrystallized from ethanol to give bright yellow crystals [7].

2.2 Synthesis of bis (benzylindene) ethylenediamine (Ben-en)

The ligand Ben-en was synthesized by refluxing an ethanolic solution of benzaldehyde (Ben) and ethylenediamine (en) (2:1 molar ratio) for one hour. The reaction mixture was cooled and a bright yellow

crystalline solid of bis (benzylidene) ethylenediamine (Ben-en) separated. The solid was filtered off and recrystallized from ethanol to give light yellow crystals [8], [9].

2.3 Synthesis of bis (benzylidene) o-phenylenediamine (Ben-opd)

The ligand Ben-opd was synthesized by refluxing an ethanolic solution of benzaldehyde (Ben) and o-phenylenediamine (opd) (2:1 molar ratio) for one hour. The reaction mixture was cooled and a bright yellow crystalline solid of bis (benzylidene) o-phenylinediamine (Ben-opd) separated. The solid was filtered off and recrytallized from ethanol to give yellow crystals.

2.4 Preparation of the complexes

The preparation of [Ni (OAcPh-en) (Ben-en)] H2O was carried out by refluxing an ethanolic solution (250m1) of Nickel diaquo complex (0.01M) with Neutral bidentate ligand bis [benzylidene] ethylenediamine (Ben-en) (0.01M) for one hour. The solution was then concentrated and cooled in air overnight. The formed crystals were collected and recrystallized, driedinair. The metal complexes of Cu(II), Co(II), Fe(II), Mn(II) and Zn(II) were prepared similarly. The mixed ligand complexes of [M (OAcPh-en) (Ben-opd)] H2O [M=Ni (II), Cu (II), Co (II), Mn(II) and Zn(II)] were also prepared similarly. The formation of the complexes may be represented by the following equations [10- 12]

OAcPh+ en H2O, OAcPh-en, Ben + en H2O Ben-en, Ben + opd, H2O Ben-opd MCl2 + 2OAcPh-en, H2O [M (H2O) 2OAcPh-en] + 2HCl [M (H2O) 2OAcPh-en] +

Ben-en 1h, Reflux [M (OAcPh-en)(Ben-en)+2H2O…(I) [M (H2O) 2OAcPh-en] + Ben-opd 1h, Reflux [M (OAcPh-en)] + 2H2O…. (II)

2.5 Analytical procedures

The complexes were analyzed for the metal contents by the EDTA-titration technique after decomposing the organic matter first with a mixture of perchloric, sulphuric and nitric acid (1:15:2.5) [13]. The infrared spectra were recorded on spectrum GX FT-IR (Perkin Elmer, USA). The magnetic susceptibility of the chelates was determined by the Gouy method at room temperature. The uv-visible spectra were recorded on Lambda 19 (Perkin Elmer, USA). The thermo gravimetric analyses were recorded on Thermo Gravimetric Analyzer (Perkin Elmer. USA). Elemental analyses were performed on a C, H, Nanalyzer (Perkin Elmer, USA). All melting points were recorded in open capillaries in a capillary melting point aaratus.

3. Results and discussion

3.1 Characterization of the complexes

The given formulae (Table 1) are based on analytical data.

Table 1 Analytical data of the complexes

Complex	Formula Weight g/mole	Colour	M.P.^{0}C	%Found.(Calc.)				% Yield	Ueff (BM)
				C	H	N	M		
[Mn(OAcPh-en) (Ben- en)]H2O	54.93	Brown	>361	69.67 (69.73)	5.88 (5.82)	9.68 (9.58)	9.32 (9.38)	76.7	5.73
[Fe(OAcPh-en) (Ben-en)]H2O	574.86	Brown	>361	69.68 (69.63)	5.77 (5.82)	9.64 (9.56)	9.47 (9.54)	74.9	5.26

[Cu(OAcPh-en)(Ben-en)]H2O	576.84	Yellowish brown	283	69.62 (69.28)	5.83 (5.78)	9.43 (9.52)	10.08 (10.1)	70.3	4.01
[Ni(OAcPh-en)(Ben-en)]H2O	577.8	Yellowish Orange	289	69.23 (69.31)	5.83 (5.78)	9.41 (9.52)	10.02 (9.98)	71.8	2.72
[Cu(OAcPh-en)(Ben-en)]H2O	587.56	Yellowish Brown	297	68.83 (68.79)	5.67 (5.73)	9.53 (9.44)	10.63 (10.71)	73.4	1.82
[Zn(OAcPh-en)(Ben-en)]H2O	587.56	Yellow	291	68.46 (68.53)	5.79 (5.72)	9.49 (9.41)	10.93 (10.97)	80.2	-
[Mn(OAcPh-en)(opd)]H2O	643.83	Brown	261	72.11 (72.03)	5.43 (5.38)	8.78 (8.83)	8.74 (8.67)	76.6	5.76
[Fe(OAcPh-en)(opd)]H2O	643.83	Brown	>361	71.86 (71.93)	5.42 (5.37)	8.87 (8.84)	8.73 (8.82)	73.3	5.23
[Co(OAcPh-en)(opd)]H2O	638.87	Brown	227	71.64 (71.58)	5.38 (5.34)	8.73 (8.78)	9.28 (9.27)	81.7	4.11
[Ni(OAcPh-en)(opd)]H2O	647.8	Yellowish Orange	222	71.54 (71.62)	5.37 (5.33)	8.87 (8.78)	9.28 (9.93)	82.4	2.68
[Cu(OAcPh-en)(opd)]H2O	643.64	Green	241	71.13 (71.06)	5.27 (5.28)	8.83 (8.74)	9.83 (9.92)	80.6	1.86
[Zn(OAcPh-en)(opd)]H2O	648.47	Yellow	247	70.82 (70.89)	5.33 (5.27)	8.77 (8.71)	10.03 (10.17)	84.4	-

3.2 Magnetic Measurements

The magnetic moment values are useful in the evaluation of results provided by the other techniques of the structural investigation [14].The magnetic moment data presented in table 1.The magnetic moment of the Cu(II) complex (1.80 - 1.85 B.M.) is very close to the spin-only value (1.73 B.M.) expected for the one unpaired electron which offers the possibility of an octahedral geometry [15]. The magnetic moment value for Fe (II) complex is 5.25-5.24 B.M. at room temperature and this value is characteristic of high spin octahedral geometry for this complex. The magnetic moment values for Co (II) complexes are 4.00-4.10 B.M. corresponding to three unpaired electrons which suggests an octahedral geometry [16]. The magnetic moment of the nickel complex at room temperature was observed 2.71-2.69 B.M. These values are in tune with a high spin configuration and show the presence of an octahedral environment around the Ni (II) ion in the complex. The Mn (II) complex show magnetic moments is 5.74-5, 75B.M. at room temperature corresponding to five unpaired electrons which suggest octahedral geometry [15]. Zn (II) complex is shown diamagnetic nature and is found to be octahedral geometry [3].

3.3 IR Spectra

The infrared spectra of the complexes have been studied to characterize their structures. The IR spectra of the complexes register v(C-O) at about 1324-1340 cm^{-1}[17]. The IR spectra of Schiff base show a sharp band near 1610 cm^{-1}which may be due to azomethin linkage and shows lowering in frequency in metal complex indicating the coordination of metal ion through azomethine linkage [18], [19]. The sharp bend in the range 750-780$cm^{-1}$and 1525- 1535 $cm^{-1}$are due to aromatic v(C-H) [20] and v(C=C) [21 respectively. The frequencies in the range 1145-1165cm^{-1}attributed to v(C-N) stretching [17] (Skoog and West, 2012). Conclusive evidence of the bonding is also shown by the observation that new bonds in the spectra of the

metal complexes at 455-460cm^{-1} and 514-525cm^{-1} these are assigned to v (M-O) and v (M-N) stretching vibrations and are not observed in the spectra of the ligand [3].

Table: 2 Infrared spectra (cm^{-1})

Complex	v(C-O)	v(C-N)	v(C-N)	v(C-C)	v(M-O)	v(M-N)
OAcPh-en	1330	1610	1144	1508	-	-
Ben-en	1324	1619	1152	1516	-	-
Ben-opd	1327	1621	1147	1513	-	-
[Mn(OAcph-en)(Ben-en)]H2O	1343	1598	1142	1534	1435	520
[Fe(OAcph-en)(Ben-en)]H2O	1335	1595	1137	1528	440	512
[Co(OAcph-en)(Ben-en)]H2O	1334	1599	1141	1529	434	515
[Ni(OAcph-en)(Ben-en)]H2O	1341	1602	1143	1532	444	508
[Cu(OAcph-en)(Ben-en)]H2O	1337	1603	1138	1534	438	510
[Zn(OAcph-en)(Ben-en)]H2O	1336	1598	1142	1532	440	517
[Mn(OAcph-en)(Ben-en)]H2O	1342	1597	1143	1534	450	519
[Fe(OAcph-en)(Ben-en)]H2O	1343	1597	1142	1527	455	512
[Co(OAcph-en)(Ben-en)]H2O	1336	1602	1137	1532	445	525
[Ni(OAcph-en)(Ben-en)]H2O	1337	1606	1143	1536	448	520
[Cu(OAcph-en)(Ben-en)]H2O	1339	1598	1142	1533	450	516
[Zn(OAcph-en)(Ben-en)]H2O	1346	1603	1144	1534	452	522

The presence of sharp band corresponding to the remaining hydroxyl group 3400cm^{-1} but it is obscured by the presence of water molecules bands. This was aeared for the most complexes and a very broad band at about 3100- 3500cm^{-1} region, which was associated with coordinated or solvent water molecules [21].

3.4 Electronic spectra

The absorption bands for the complexes will help to give an idea of their structure [22]. Electronic spectrum of Co(II) complex exhibits absorption bands at 12,920, 16,260, and 23,450 cm^{-1}, which may be assigned to $^4T_1$ (F) $^4T_2$ (F), $^4T_1$ (F) $^4A_2$ (F) and $^4T_1$ (F) $^4T_1$ (P) transitions respectively which are in support of octahedral arrangement of binding centres around the metalion [20]. The electronic spectra of Mn (II) complexes show the absorption bands in the range 16970-19540, 22280-24390 and 26109-27624cm^{-1}. These absorption bands may be assigned to the ^{6}A1g 4A1g (^{4}G), ^{6}A1g ^{4}A2g (^{4}G), and $^6_{A_{1g}}$ ^{4}Eg, ^{4}A1g (^{4}G) transitions, respectively. These bands suggest that the complexes possess an octahedral geometry [19].Bandsattheregions675nm(14815cm^{-1}) and 535nm (18690cm^{-1}) for Cu(II) complex were assigned to the ^{2}B1g ^{2}B2g and ^{2}B2g^{2}Eg transitions respectively, found for octahedral Cu(II)complexes[23].The electronic spectrum of the Fe(II) complex exhibited and at 11,200cm^{-1} assigned to the ^{5}T2g^{5}Eg transition. A strong charge transfer band is observed at 26,000cm^{-1}. These data suggest an octahedral geometry [24]. The electronic spectra of the Ni (II) complex showed d-d bands in the region 10115 and 26280-26410cm^{-1}^{3}A2g

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(F) $^{3}T2g$ (F) and 3A2g (F) $^{3}T2g$ (P) respectively, consistent with their well-defined octahedral configuration. The Zn (II) complex has not shown any d-d absorptions [15].

3.5 Thermo Gravimetric Analysis

The analysis of the thermal curve of the complexes clearly indicated that the weight loss between 35-105^{0}C corresponds to one water molecule for all complexes. Because of the low temperatures, this molecule may be considered as crystal water [6].

Table 3: Antimicrobial activity

Complexes	**Diameter of inhibition zone (mm)**	
	Bacillus Substlis	***E.Coli***
[Mn(OAcPh-en)(Ben-en)]H2O	26	These metal complexes do not have effect on *E.Coli* bacteria so there is no inhibition zone.
[Fe(OAcPh-en)(Ben-en)]H2O	23	
[Co(OAcPh-en)(Ben-en)]H2O	27	
[Ni(OAcPh-en)(Ben-en)]H2O	19	
[Cu(OAcPh-en)(Ben-en)]H2O	22	
[Zn(OAcPh-en)(Ben-en)]H2O	27	
[Mn(OAcPh-en)(Ben-en)]H2O	23	
[Fe(OAcPh-en)(Ben-en)]H2O	22	
[Co(OAcPh-en)(Ben-en)]H2O	23	
[Ni(OAcPh-en)(Ben-en)]H2O	18	
[Cu(OAcPh-en)(Ben-en)]H2O	19	
[Zn(OAcPh-en)(Ben-en)]H2O	24	

The curves in the range between 105-385^{0}C suggested that loss in weight for all complexes correspond to evaporation of bis [benzylidene] ethylenediamine (Ben-en) and bis [benzylidene] o-phenylinediamine (Ben-opd). The range above 385^{0}C loss in weight correspond to remaining organic ligand molecules. In all cases, final products are metal oxides. These results are good and in accordance with the composition of the complexes.

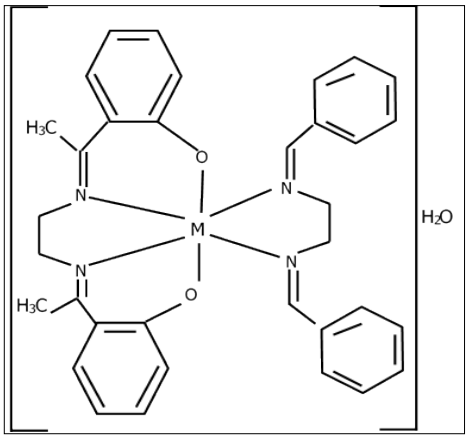


Figure 1 [M (OAcPh-en) (Ben-en)] H2O [M=Ni(II), Cu(II), Co(II), Fe(II), Mn(II) and Zn(II)]

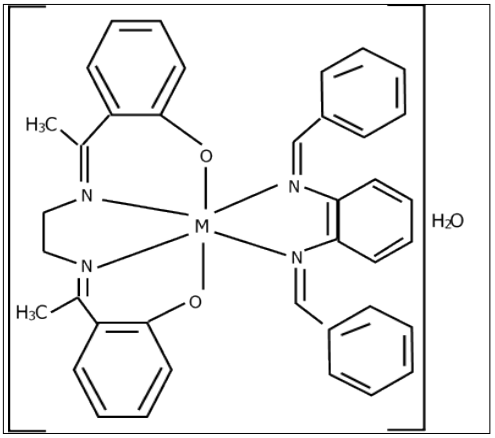


Figure 2 [M (OAcPh-en) (Ben-en)] H2O [M=Ni(II), Cu(II), Co(II), Fe(II), Mn(II) and Zn(II)]

3.6 Antimicrobial activity

The antimicrobial activity of the complexes against Gram positive bacteria *Bacillus Substlis* and Gram negative bacteria *E. Coli* have been done using the "Agar diffusion method" [24], [25]. The diameter of inhibition zone of the various compounds is recorded in table III. All the compounds have significant antibacterial activity at $1.0 \times 104\mu gm]^{-1}$ against bacteria. The compounds [M(OAcPh-en) (Ben-en)]. H_2O,[M=Ni(II), Cu(II), Co(II), Fe(II), Mn(II) and Zn(II)] are more active as compared to the compounds [M (OAcPh-en) (Ben-opd)]. H_2O, [M=Ni (II), Cu (II), Co (II), Fe (II), Mn(II) and Zn(II)] against bacteria. All the compounds screened are more active against gram positive bacteria *Bacillus Substlis* then gram negative bacteria *E.Coli*., [7], [26].

4 Conclusion

In this study, it is observed that the complexes of the mixed ligands complexes have excellent antimicrobial potentials at different concentrations, against gram positive bacteria, *Bacillus subtilis,* hence their activities against this strain of microorganism increased with increased in concentration. For gram negative bacteria, Escherichia coli, it is observed that,in all concentrations there is no indication of inhibition diameter zones, it therefore means that the pathogen, *E. Coli* is being reported as resistant to the antimicrobial potential of the mixed ligand complexes.

Compliance with ethical standards

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Disclosure of conflict of interest

The authors declare that there is no conflict of interest

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